PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application: Silverberg et al.	)	Group Art Unit: 1755
Serial No. 09/955,644	)	Examiner: Isis A. D. Ghali
Filed: September 18, 2001	)	Atty. Docket No. 1893

For: NON-REACTIVE ADHESIVE USEFUL IN TRANSDERMAL DRUG DELIVERY SYSTEMS

DECLARATION UNDER 37 C.F.R. § 1.131

Commissioner for Patents
Alexandria, VA 22313-1450

Sir:

We, Eric Silverberg, Rama Chandran, Paul Foreman, Michael Philbin and Smita Shah, hereby declare:

That we are the inventors of the subject matter claimed in subject application Serial No. 09/955,644, which application claims the benefit of the earlier filing date of our provisional application No. 60/234,248, filed September 19, 2000.

That we have read, and understand, the Office actions and references applied therein, including the Tan et al. patent (U.S. Patent No. 6,077,527), and understand that the Examiner has rejected the claims in view of the Tan et al. patent disclosure.

That the attached document is a copy of an invention disclosure record, which document was prepared and executed prior to the June 20, 2000 publication date of Tan et al. and evidences that the claimed invention was made prior to the publication of the Tan et al. patent.

We further declare that all statements made herein of my own knowledge are true and that all statement made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by a fine or imprisonment or both under 1001 of Title 18 of the United States Code and such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

2/23/2006
Date

Eric Silverberg

2/21/2006
Date

Rama Chandran

2/23/2006
Date

Paul Foreman

2/22/2006
Date

Michael Philbin

2/23/2006
Date

Smita Shah

[redacted]

A

INVENTION DISCLOSURE

TITLE: NON-REACTIVE ACRYLIC PRESSURE SENSITIVE ADHESIVES FOR TRANSDERMAL DRUG DELIVERY APPLICATIONS

I. The Invention

A. Description of the Invention

This invention relates to pressure sensitive adhesive composition for use in transdermal drug delivery systems in which the adhesive composition is not crosslinked and is chemically inactive to the active (drug and excipient) ingredients contained in the transdermal formulation.

B. Purpose of the Invention

The purpose is to prepare a pressure sensitive adhesive that does not chemically react with the active (drug and excipient) ingredients contained in a transdermal formulation and maintains its adhesive properties without the need for crosslinking. No chemical reaction is meant to be no covalent bonds formed or broken between the active ingredients in the transdermal patch and/or the polymer (backbone or side group).

Current adhesives are copolymers that are comprised of functional monomers that are potentially reactive to the active ingredients. Functional monomers are meant to include but not limited to carboxylic acid, hydroxyl, epoxy, and acetate functionality e.g. acrylic acid, methacrylic acid, 2-hydroxyethyl acrylate, 2-hydroxyethyl methacrylate, vinyl acetate, glycidal methacrylate.

The sustained release of a pharmaceutically active drug to the skin of a human patient is of great importance. Transdermal drug delivery systems have been developed that offer this key characteristic. However, the reactivity of active ingredients within a transdermal drug delivery system with the polymer backbone or side group and with residual monomer is a threat to sustained release. Transdermal patch manufacturers are limited by this reactivity. In addition, they have excluded chemically sensitive, highly reactive drugs from their product offerings. Transdermal patch manufacturers have partially engineered around this problem by constructing the patch supersaturated with the drug. This method maintains a constant drug flux regardless of any interaction between the drug and reactive agents. This is very wasteful in that approximately only 10% of the drug in the

patch eventually enters the bloodstream. The method is economically acceptable with less expensive drugs however this method becomes cost prohibitive when used with more expensive drugs.

A second area of concern to transdermal patch formulators is the formation of new compounds within the patch as a result of a chemical reaction between the active ingredients and the adhesive. These new compounds may upset the flux of the drug. In addition, the new compound may be pharmaceutically active in the body and cause deleterious effects.

C. Attachments

1. Tables 1 shows the adhesive properties compared with functional acrylic and vinyl-acrylic adhesives. In addition, potential drug/excipient interactions with monomer/polymer are shown.
2. Laboratory notebook pages: pp. 10634-143,144; 11231-7,8,35,36,58,59; 10872-48, 49.

D. Preferred Embodiment

The following adhesive (copolymer) composition gives the best performance (adhesive) and best performance (non-reactive, assumed as of this writing)

% by weight	Monomer
45	2-Ethylhexyl acrylate
35	Methyl acrylate
20	N-substituted acrylamide (t-Octyl acrylamide preferred)

The final adhesive is not formulated with any crosslinker. The synthesis of this material is of equal importance to the composition. The process is such to give molecular weights >600,000 Mw.

II. Literature Search Details

B. Groff conducted a literature search. The following databases were searched with the start year in ( ): CAPlus (1967), HCAPlus (1967), IPA (1970), IFICAT (1950), JAPIO (1976), WPIDS (1963), and Rapra (1972). Relevant literature includes:

US 4655767
USRE035474
US5186938

III. Inventorship

Eric Silverberg, Rama Chandran, Paul Foreman, Michael Philbin, Smita Shah

IV. Dates and Proof of Conception and Reduction to Practice

Project initiation worksheet written on [redacted]
First technical discussions occurred at a meeting on [redacted]
First reactions carried out on [redacted]

V. Means to Detect Infringement

Nuclear Magnetic Resonance (NMR) spectroscopy and Fourier-Transform Infrared spectroscopy (FT-IR) for compositional analysis of adhesive.

VI. Disclosures

None

VII. Marketing Manager and SBU

Ellen Greenhorn, Transdermal Adhesives

VIII. Signatures and Comments

Inventors:

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Witness: Read and understood by:
_____
Dated: _____

Technical Director:
_____
Dated: [redacted]

Comments:

New project

ORIGINAL Notebook

№ 10634-143

Project No.

Date Started [redacted]

Object High Performance Monoacrylic PSA for TDD. Prepare Hmw polymer (~400,000 Mw) that can accept large amount of enhancers (~40%). Need a polymer that is non-reactive with the enhancers, also with high performance.

[redacted]

High Performance Nonreactive PSA for TDD [redacted]

A=control, 2-EHA 62.23/ MA 32.02/ AA 5.73/ GMA 0.029 and B, C=2-EHA 60/ MA 40

	A		B		C	
Initial Charge:	pphm	wt (g)	pphm	wt (g)	pphm	wt (g)
2-Ethylhexyl acrylate	46.39	231.95	44.73	223.65	44.73	223.65
Methyl acrylate	23.58	117.9	29.46	147.3	29.46	147.3
Acrylic acid	4.31	21.55	-	-	-	-
Glycidyl methacrylate	0.029	0.145	-	-	-	-
Ethyl acetate	43.39	216.95	43.39	216.95	43.39	216.95
Hexane	9.81	49.05	9.81	49.05	-	-
Acetone	-	-	-	-	9.81	49.05
Initial Initiator-I:						
Ethyl acetate	21.25	106.25	21.25	106.25	21.25	106.25
AIBN	0.016	0.08	0.016	0.08	0.016	0.08
Initial Initiator-II:						
Ethyl acetate	4.74	23.7	4.74	23.7	4.74	23.7
AIBN	0.019	0.095	0.019	0.095	0.019	0.095
Initiator SA:						
Ethyl acetate	13.13	65.65	13.13	65.65	13.13	65.65
AIBN	0.13	0.65	0.13	0.65	0.13	0.65
Monomer SA:						
2-Ethylhexyl acrylate	15.84	79.2	15.27	76.35	15.27	76.35
Methyl acrylate	8.44	42.2	10.54	52.7	10.54	52.7
Acrylic acid	1.42	7.1	-	-	-	-
Ethyl acetate	11.28	56.4	11.28	56.4	11.28	56.4
Solvent SA:						
Ethyl acetate	8.05	40.25	8.05	40.25	8.05	40.25
Hexane	12.48	62.4	12.48	62.4	-	-
Acetone	-	-	-	-	12.48	62.4
Total weight	224.3	1121.5	224.3	1121.5	224.3	1121.5
Total solid	100.174	500.87	100.174	500.87	100.174	500.87
Theoretical %solid	44.66		44.66		44.66	
	44.7		43.7		43.8	43.6

Comments: Eval: Pg = 144

Apparatus: 2L RB flask, ss stirrer, thermometer, condenser, water bath, SA funnels

Procedure:

1. Charge IC to flask, add initial initiator-I to the flask.
2. Start agitation, heat it to reflux.
3. Hold at reflux for 10 min and add initial initiator-II to the flask.
4. At reflux + 45 min, start monomer SA over 1 h, while maintaining reflux.
5. At reflux + 45 min, start initiator SA over 1 h, while maintaining reflux.
6. At reflux + 190 min (3h 10min), start solvent SA over 3 h.
7. Hold at reflux for 6.5 h——can be split.
8. Cool to room temp. Discharge in an ppropriately labeled container.
9. Analysis: %Solids, BFV, GPC, Resi. monomers, IV (Tol.)

Submit A-C on [redacted]

Conclusion: Sl. exotherm (~2°C) is present when initial initiator II is added. Exotherm lasts 30-45 secs. Final lacquers are clear. Reaction 143C with Acetone has a higher mw than control and 143B.

Eval: PSLA (Target Lc) reports sample 143A (control) meets pred specs. Sample 143B,C did not meet spec.

[redacted]

Nº 10634-144

Project No. ______ Date Started [redacted]

Object: Continue from Pg # 10634-143. Comments + Eval.

Comments

A) - RT - (RPM = 200) - Add Initial initiator #1 and start heat
- RT + 20 mins (76.5/84 °C) - Reflux (1½ hours) no EXO
* - R + 10 mins (73/82 °C) - Add Initial initiator #2 . Exotherm ~2 °C which lasts ~30 secs. (Foamy) Exo not visible at low H_2O bath level
- RT 45 mins (70/82 °C) - Flat mix mass (RPM = 300) Start SA_1, SA_2
- R + 1 HR/45 mins (72.5/90 °C) - Complete SA_1, SA_2 - Flat mix mass
- Complete reaction as stated in procedure.
- Before cooling (72/91 °C) - Flat mix mass (RPM = 300)
- * Final lacquer at room temp is clear

B) Like "A" except reflux temp = (76.5/84 °C)

C) Like "A" except more viscous than "A" and "B".
Note: ~1" S. climb before SA_3 (RPM = 300).
Final lacquer at (72.5/93 °C) (RPM = 300) has ~½" S. climb

Eval:

	143A	143B	143C	
% solids	43.4	43.8	43.6	see Pg for BFV # 152 study
BFV (cps)	35,200 ~~35,000~~ 9/14/99	27,500 ~~25,500~~ 9/29	23,500 ~~[illegible]~~ 9/29	
IV ~~[illegible]~~	0.63 0.84	1.38 1.40	1.68 1.67	
IV (Final)	0 6.1	2.4 5.9	0 9.5	IV in toluene IV bath = 30.2 °C
GPC (THF) Mw	5.24E5	4.98E5	6.57E5	IV (viscometer) # NS22 (Sup ~~[illegible]~~ 2353) - 4.84E5
Mn	4.58E4	4.72E4	4.69E4	5.07E4
Disp	11.43	10.61	14.02	See Pg # 150 for GPC ... DMAC 9.47

GC (ppm) — % solids 40.0

				2353
MA	2890, 2935	3265, 3260	3450, 3445	2850, 2855
2EHA	5500, 5645	4850, 4830	4305, 4305	9685, 9620
AA	555, 650	~~[illegible]~~ -	~~[illegible]~~ -	885, 910

WORK OF: [signature] DATE [redacted]

NATIONAL STARCH

Nº 11231- 7

Project No. Ref: 10634-149 Date Started [redacted]

Object High Performance Nonreactive PSA for TDD. Improve performance

	A=BA 50/MA 50		B=EHA 60/MA 35/MMA 5		C=EHA 60/MA 35/OA 5	
Initial Charge:	pphm	wt (g)	pphm	wt (g)	pphm	wt (g)
2-Ethylhexyl acrylate	-	-	44.73	223.65	44.73	223.65
Methyl acrylate	38.83	184.15	25.78	128.9	25.78	128.9
Butyl acrylate	37.28	186.4	-	-	-	-
Methyl methacrylate	-	-	5	25	-	-
I-OA	-	-	-	-	5	25
Ethyl acetate	64.64	323.2	64.64	323.2	64.64	323.2
Acetone	9.81	49.05	9.81	49.05	9.81	49.05
AIBN	0.016	0.08	0.016	0.08	0.016	0.08
Initial Initiator-II:						
Ethyl acetate	4.74	23.7	4.74	23.7	4.74	23.7
AIBN	0.019	0.095	0.019	0.095	0.019	0.095
Initiator SA:						
Ethyl acetate	13.13	65.65	13.13	65.65	13.13	65.65
AIBN	0.13	0.65	0.13	0.65	0.13	0.65
Monomer SA:						
2-Ethylhexyl acrylate	-	-	15.27	76.35	15.27	76.35
Methyl acrylate	13.18	65.9	9.22	46.1	9.22	46.1
Butyl acrylate	12.73	63.65	-	-	-	-
Ethyl acetate	11.28	56.4	11.28	56.4	11.28	56.4
Solvent SA:						
Ethyl acetate	8.05	40.25	8.05	40.25	8.05	40.25
Acetone	12.48	62.4	12.48	62.4	12.48	62.4
Total weight	224.3	1121.5	224.3	1121.5	224.3	1121.5
Total solid	100.174	500.87	100.174	500.87	100.174	500.87
Theoretical %solid	44.66	223.3	44.66	[redacted]	44.66	[redacted]

Submitted

Comments + Eval: for # 11231-08

Apparatus: 2L RB flask, ss stirrer, thermometer, condenser, water bath, SA funnels

Procedure:

1. Charge IC to flask.
2. Start agitation, heat it to reflux.
3. Hold at reflux for 10 min and add initial initiator-II to the flask.
4. At reflux + 45 min, start monomer SA over 1 h, while maintaining reflux.
5. At reflux + 45 min, start initiator SA over 1 h, while maintaining reflux.
6. At reflux + 190 min (3h 10min), start solvent SA over 3 h.
7. Hold at reflux for 6.5 h——can be split.
8. Cool to room temp. Discharge in an ppropriately labeled container.
9. Analysis: %Solids, BFV, GPC, Resi. monomers, IV

Conclusion: Reactor 07B has high level of foam after adding Initiator II. Hot water bath had to be removed. Foam dispersed in 15 secs. Reaction is very similar in visc to 10634-143C (2EHA=60/MA=40). Final lacquer is clear & pourable at RT.

Reactor 07A containing 5 pts of MMA has a lower Mw than 11231-07B. Final lacquer is clear & pourable at RT.

* Both reactions have ~ 2°C exotherm after adding Initiator II. Exo lasts ~ 45 secs.

WORK BY: [redacted] DATE: [redacted]

NB 11631- 8

Project No.

Date Started [redacted]

Object Continue from Pg # 11231-7. Comments · Eval

Comments:

A) 79.5/86 °C B) 74.5/87 °C - Reflux (Rpm=200) No exo - 1 ohne in condenser
- R+10 min, All in 4" 2 A) Lt visc B) Mod/Hi
A) ~2 °C Exo, lasts 45 secs (Rpm=240)
* B) " "
High foam - Drop H_2O bath (...
A) 77/92 °C B) 71.5/90 °C - R+45 min A) mod visc, deep vortex (Rpm=290) - Foam lasts ~15 secs
* A) " B) Flat air mass
Mod/High visc (Erpm) - Did not drop H_2O bath
A) 77/88 °C B) 73/92 °C - R+1Hr/45 min, Complete SA_1 · SA_2
Flat air mass B>A (Rpm=300).
A) 74.5/90 °C B) 73.5/90 °C - R+3Hr/10 min A) Flat B) ~1/2" s.c. } Start SA
Complete reactions as stated in ~~proc~~ procedure
AB) 70/84 °C - Cool, Both reactions are flat air mass
- Lacquers are clear

EVAL:	07A	07B	2353	10634 133C		07A
1. Solids	44.4	44.5	40.0	43.6	% solids	34.9
BEv(cps)	15,180	58,600		65,500	BEv(cps)	316c
Iv(Tol)	1.29	1.55	1.13	1.63		
% Ensol	4.4	0.4	0	2.8		

Lg # [redacted]

Rs (ppm)

Previous GPC in THE was not reproducible

	07A	07B	2353	10634
mA	2995	3455	—	—
mmA	32	TOA = 290	—	—
2EHA	4665	5170	—	

GPC (mw in DMAC, PVA column)

	07A	07B	2353	10634
Mw	283,109	316,502	247,584 / 302,357	311,284 / 323
Mn	24,926	25,064	28,307 / 29,837	20,957 / 24
Disp	11.36	12.63	10.53 / 10.13	14.85 / 13

WORK OF: [signatures]

NATIONAL STARCH

Nº 11231- 35

Project No. Ref: 11231-78 Date Started [redacted]

Object High Performance Mix Reactive PSA for TDD.

1. 2EHA= 57.5/ MA= 32.5/ ToA = 10 B) 2EHA= 52.5 / MA= 27.5/ ToA= 20

Objective: To observe an effect on perf..mance improvement w/t-OA ladder; Refer 11231-78

	Theo. Tg -42.88°C A=EHA 57.5/MA 32.5/t-OA 10		Theo. Tg -35.65°C B=EHA 52.5/MA 27.5/t-OA 20	
Initial Charge:	pphm	wt (g)	pphm	wt (g)
2-Ethylhexyl acrylate	42.23	211.15	37.23	186.15
Methyl acrylate	23.28	116.4	18.28	91.4
t-OA	10	50	20	100
Ethyl acetate	64.64	323.2	64.64	323.2
Acetone	9.81	49.05	9.81	49.05
AIBN	0.016	0.08	0.016	0.08
Initial Initiator-II:				
Ethyl acetate	4.74	23.7	4.74	23.7
AIBN	0.019	0.095	0.019	0.095
Initiator SA:				
Ethyl acetate	13.13	65.65	13.13	65.65
AIBN	0.13	0.65	0.13	0.65
Monomer SA:				
2-Ethylhexyl acrylate	15.27	76.35	15.27	76.35
Methyl acrylate	9.22	46.1	9.22	46.1
Ethyl acetate	11.26	56.4	11.26	56.4
Solvent SA:				
Ethyl acetate	8.05	40.25	8.05	40.25
Acetone	12.48	62.4	12.48	62.4
Total weight	224.3	1121.5	224.3	1121.5
Total solid	100.174	500.87	100.174	500.87
Theoretical %solid	44.66	223.3	44.66	

Sub. (1, 3 4s) [redacted]

Comments Excl: Ref = 11231-36

Apparatus: 2L RB flask, ss stirrer, thermometer, condenser, water bath, SA funnels

Procedure:

1. Charge IC to flask.
2. Start agitation, heat it to reflux.
3. Hold at reflux for 10 min and add initial initiator-II to the flask.
4. At reflux + 45 min, start monomer SA over 1 h, while maintaining reflux.
5. At reflux + 45 min, start initiator SA over 1 h, while maintaining reflux.
6. At reflux + 190 min (3h 10min), start solvent SA over 3 h.
7. Hold at reflux for 6.5 h—can be split.
8. Cool to room temp. Discharge in an ppropriately labeled container.
9. Analysis: %Solids, BFV, GPC, Resi. monomers, IV

Conclusion: Reactor 11231-35A has high level of foam 5 mins after adding initiator II. Drop hot H_2O bath several times to control foam. There was no exotherm. Foaming lasted ~ 3 mins. Reactor 35B has moderate foaming.

PSCA (T.L.) reports 11231-35B has significant improvement on cohesion, along with good tack, peel and shear on stainless steel.

[redacted]

NY 11231-35

Date Started [redacted]

Continue from Pg # 11231-35. Comments = Eval

Comments:

AB) RT - Start heat (RPM = 200)

A) 74/87°C B) 81.5/86°C Reflux (1 ml/min in condenser)

A) 75/88°C B) 80.5/87°C R+10 mins, 4 I/min vac (RPM = 240) ADJ

Init II. ~1°C Exotherm

* A) R+15 mins, High foam level. Drop H_2O bath 2 times to control high level of foam - NO EXO

A) 73/86°C B) 76/87°C, R+45 mins - Flat air mass (A=B) (RPM = Start SA_1 : SA_2

A) 74/91°C B) 76.5/92°C, R+1 Hr/45 mins - Complete SA_1 : SA_2 (RPM Flat air mass A=A (Reflux ~1/2 obs)

Complete reaction as stated in procedure

* Final Isocyanates are clear.

Eval:	35A	35B	11231-7B	10634-143C
% Solids	43.2	44.2	44.5	43.6
IV (70C)				
Insol				
[redacted] BEW (g/eq)	25,700	16,250	54,600	65,800
Res (ppm)				
MA	3075 4985	2810/2600	3275	3355/3300
2EHA	5020/5010	5020/5010	5275	4700/4640
ToA	470	580/585	240	—

GPC:

Analytical conditions:
Column: Asahipak GF-7M HQ(7909043) Mobile phase: DMAC+0.03M NaNO3
Injector: Waters WISP 717plus Column Temp.: 60C
Detector: RID-6A Standard: PS
Pump: Waters 515 Calibration: Linear(LI21)
Injection:100 ul Flow rate: 1 ml/min.
Injection No: 2 Sample Conc.:~20 mg/4 ml
End of calculation:491 Run time: 17 minutes
Processing program: P.E. Nelson Turbochrom SEC versi[redacted]

MOLECULAR WEIGHT DISTRIBUTION AVERAGES

SAMPLE	Mw	Mn	Mw/Mn
10634-143C	575307	45321	12.69
11231-7B	547305	46607	11.64
11231-35A	485658	45698	10.63
11231-35B	403698	47080	8.57

Note: [redacted] new column was used. Results of N3C + 7B vary from previous work. Will use these new GPC results.

DATE: [redacted]

№ 11231- 58

Project No. Ref: 11231-358, 11231-52 **Date Started** [redacted]

Object High Performance Non-reactive PSA for TDD. Experimental study regarding Tg

358=2-EHA 52.5/MA 27.5/ I-OA 20

Theo. Tg: 358=-35.7, A=-20, B=-36, C=-27, D=-29, E=-42

	358	A		B		C		D		E	
Initial Charge:	pphm	pphm	wt (g)	pphm	wt (g)	pphm	wt (g)	pphm	wt (g)	pphm	wt (g)
2-Ethylhexyl acrylate	37.23	33.68	168.4	39	195	31.91	159.55	31.91	159.55	42.55	212.75
Methyl acrylate	18.28	14.96	74.8	13.29	66.45	19.94	99.7	23.27	116.35	13.29	66.45
I-OA	20	30	150	25	125	25	125	20	100	20	100
Ethyl acetate	64.64	64.64	323.2	64.64	323.2	64.64	323.2	64.64	323.2	64.64	323.2
Acetone	9.81	9.81	49.05	9.81	49.05	9.81	49.05	9.81	49.05	9.81	49.05
AIBN	0.016	0.016	0.08	0.016	0.08	0.016	0.08	0.016	0.08	0.016	0.08
Initial Initiator-II:											
Ethyl acetate	4.74	4.74	23.7	4.74	23.7	4.74	23.7	4.74	23.7	4.74	23.7
AIBN	0.019	0.019	0.095	0.019	0.095	0.019	0.095	0.019	0.095	0.019	0.095
Initiator SA:											
Ethyl acetate	13.13	13.13	65.65	13.13	65.65	13.13	65.65	13.13	65.65	13.13	65.65
AIBN	0.13	0.13	0.65	0.13	0.65	0.13	0.65	0.13	0.65	0.13	0.65
Monomer SA:											
2-Ethylhexyl acrylate	15.27	13.82	69.1	16	80	13.09	65.45	13.09	65.45	17.45	87.25
Methyl acrylate	8.22	7.54	37.7	6.71	33.55	10.06	50.3	11.73	58.65	6.71	33.55
Ethyl acetate	11.28	11.28	56.4	11.28	56.4	11.28	56.4	11.28	56.4	11.28	56.4
Solvent SA:											
Ethyl acetate	8.05	8.05	40.25	8.05	40.25	8.05	40.25	8.05	40.25	8.05	40.25
Acetone	12.48	12.48	62.4	12.48	62.4	12.48	62.4	12.48	62.4	12.48	62.4
Scavenger SA:											
Ethyl acetate	5	5	25	5	25	5	25	5	25	5	25
t-APP (75%)	[illegible]	0.7	3.5	0.7	3.5	0.7	3.5	0.7	3.5	0.7	3.5
Total weight	224.3										
Total solid	100.174										
Theoretical %solid	44.66										

Apparatus: 2L RB flask, ss stirrer, thermometer, condenser, water bath, SA funnels

Procedure:

1. Charge IC to flask.
2. Start agitation, heat it to reflux.
3. Hold at reflux for 10 min and add Initial Initiator-II to the flask.
4. At reflux + 45 min, start monomer SA over 1 h, while maintaining reflux.
5. At reflux + 45 min, start initiator SA over 1 h, while maintaining reflux.
6. At reflux + 190 min (3h 10min), start solvent SA over 3 h
7. Hold at reflux for 6.5 h—(no spill.
8. - Add over 2 h - HN , hr
9. Cool to room temp. Discharge in an appropriately labeled container.
10. Analysis: %Solids, BFV, GPC, Resid. monomers, IV

Submitted A-E) [redacted]

11231
58

Comments: Goal: [illegible]

[redacted]

GPC:

Conclusion: Reactors D·E have high level of foam. Drop H_2O bath for ~30 s
Reactors A-C have mod foam level. Did not drop H_2O bath. PSCA reports
samples 358C + 58D appear the best with peel values about cov:lin and shear C4p. 5k

WORK OF: / [signature] / [signature]

NATIONAL STARCH Nº 11231- 59

roject No. Date Started [redacted]

bject Continue from Pg # 11231-58, Comments

Comments:

A-E) 80/84°C - Reflux (RPM=220). R=1-1½ actives. No Exo

A) 79.5/85°C - R+10 mins, 47/mL visc (RPM=335) - Add init II

" 75.5/85°C - R+20 mins Foam (~1°C Exo) - Did not drop but H_2O bath. Exo lasts 30 secs - 1 min.

" 74.5/84°C - R+45 mins, Flat air mass (RPM=300) Start SA_1 · SA_2

" Complete reaction as stated in procedure

" 71/87°C - Complete reaction (RPM=300) - Flat air mass - cool

B) Like "A" except R+20 mins: Foam (~2°C Exo) Exo lasts ~1 min

C) Like "A" except R+20 mins: Foam (~2°C Exo) Exo lasts ~1 min

D) Like "A" except R+20 mins: Foam (~2°C Exo) Drop hot H_2O bath 30 secs later foam is moderate but controllable. Apply hot H_2O bath

E) Like "A" except R+20 mins: Foam (~2°C Exo) Drop hot H_2O bath 30 secs later foam is moderate but controllable. Apply hot H_2O bath.

VAL:	58A	58B	58C	58D	58E	Control 11231-35B
% Solids	43.8	43.9	43.7	43.5	44.0	44.2
BFV (cps)	6940.0	6950.0	12,380.0	21,450.0	12,920.0	16,250.0
(ppm)						
mA	335	285	405	575	165/165	2565/2465
2EHA	400	465	335	450	385/420	3920/4250
TOA	260	145	185	135	125/130	655/795

J. Milk / Instructor [redacted]

LN. 10067-40 [illegible]

Project No. Date Started [redacted]

Object Non Reactive High Performance PSA

Coated samples 11231-58A, 58B, 58C, 58D, and 58E on liner at 1 dry mil then laminated to 2 mil polyester.

Ran performance testings. Result is followed:

[redacted] T.Le)

Non Reactive High Performance PSA for TDD

	Solvent	2-EHA	MA	TOA	Theory Tg(C)
11231-58A	Acetone/EtOAc	47.5	22.5	30	28 C
11231-58B	Acetone/EtOAc	55	20	25	36 C
11231-58C	Acetone/EtOAc	45	30	25	27 C
11231-58D	Acetone/EtOAc	45	35	20	29 C
11231-58E	Acetone/EtOAc	60	20	20	42 C

Performance on S.S panels

Sample ID	11231-58A	11231-58B	11231-58C	11231-58D	11231-58E
Solids	43.8%	43.9%	43.7%	43.5%	44.0%
Viscosity	6,940cps	6,950cps	12,380cps	21,450cps	12,920cps
Coating Weight	17.1#/r	17.3#/r	17.3#/r	17#/r	18.1#/r
Peel,initial 20min.@RT (oz/in)	46,41,41 (zipped)	58,61,60 (af)	65,56,58 (af)	54,59,61 (af)	58,59,59 (af)
Avg	43	59	69	57	59
Peel,24hrs OP@RT (oz/in)	71,73,75 (af)	68,67,76 (af)	63,69,71 (af)	59,54,64 (af)	66,69,56 (af)
Avg	73	70	68	59	64
Peel, 1Wk OP@RT (oz/in)	67,70,63 (af)	63,61,65 (af)	60,60,60 (af)	58,54,54 (af)	68,67,60 (af)
Avg	67	63	60	56	64
Shear, 4PSI@RT(hr)	6.0,6.2,6.3 (cf)	1.2,1.3,1.3 (cf)	5.7,5.8,6.0 (cf)	4.8,5.1,5.2 (cf)	0.4,0.4,0.5 (cf)
Avg	6.2	1.3	5.8	5	0.4

Note:

(af): adhesive failure

(cf): cohesive failure

WORK OF: Tuyet Le DATE: [redacted]

I WITNESS THIS DOCUMENT AND UNDERSTAND ITS CONTENTS

NATIONAL STARCH

Nº 10872- 49

Project No.

Date Started [redacted]

Object Non Reactive High Performance PSA

Coated samples 11231-60, 64A, 64B on lines at 1 dry mil then transferred to 2 mil PET.

Ran performance testings.

Result is followed:

[redacted](T.Le)

Non Reactive High Performance PSA for TDD

		2-EHA	MA	TOA
11231-60	Higher MW of 35B	52.5	27.5	20
11231-64A	Same as 58C	45	30	25
11231-64B	Higher MW of 58C	45	30	25
11231-58C	Control	45	30	25

Performance on S.S panels

Sample ID	11231-58C	11231-60	11231-64A	11231-64B
Solids	43.7%	47.6%	43.4%	43.7%
Viscosity	12,380cps	31,850 cps	14,380 cps	24,250 cps
Coating Weight	17.3#/r	18.5#/r	18.2#/r	18.1#/r
Peel,initial 20min.@RT (oz/in)	65,56,58 (af)	53,54,53 (af)	52,38,26 (hz)	36,57,40 (hz)
Avg	69	53	39	44
Peel,24hrs OP@RT (oz/in)	63,69,71 (af)	61,59,60 (af)	61,61,61 (af/lz)	53,56,61 (af/lz)
Avg	68	60	61	57
Peel, 1Wk OP@RT (oz/in)	60,60,60 (af)	63,62,64 (af)	69,69,65 (af)	69,69,61 (af)
Avg	60	63	68	66
Shear, 4PSI@RT(hr)	5.7,5.8,6.0 (cf)	1.7,1.8,2.0 (cf)	7.7,8.6,7.6 (cf)	9.2,9.2,9.9 (cf)
Avg	5.8	1.8	8	9.4

WORK OF: [redacted] DATE: [redacted]